Revision 1

Joegoldsteinite: A new sulfide mineral ($MnCr_2S_4$) from the IVA iron meteorite, Social Circle

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ABSTRACT

Joegoldsteinite, a new sulfide mineral of endmember formula $MnCr_2S_4$, was discovered in the Social Circle IVA iron meteorite. It is a thiospinel, the Mn analogue of daubréelite ($Fe^{2+}Cr_2S_4$), and a new member of the linnaeite group. Tiny grains of joegoldsteinite were also identified in the Indarch EH4 enstatite chondrite. The chemical composition of the Social Circle sample by electron microprobe is (wt%) S 44.3, Cr 36.2, Mn 15.8, Fe 4.5, Ni 0.09, Cu 0.08, total 101.0, giving rise to an empirical formula of $(Mn_{0.82}Fe_{0.23})Cr_{1.99}S_{3.95.}$ The crystal structure, determined by electron backscattered diffraction, is a *Fd3m* spinel-type structure with a = Å, V = 1033.4 Å^3, *and* Z = 8.

Keywords: Joegoldsteinite, $MnCr_2S_4$, new sulfide mineral, thiospinel, Social Circle IVA iron meteorite, Indarch EH4 enstatite chondrite

INTRODUCTION

Thiospinels have a general formula of AB_2X_4 where A is a divalent metal, B is a trivalent metal and X is a -2 anion, typically S, but in some cases Se or Te. Some thiospinels are magnetic semiconductors and have been studied extensively by materials scientists. Synthetic $MnCr_2S_4$ is known to be a ferrimagnetic insulator (Menyuk et al. 1965; Darcy et al. 1968; Lotgering 1968; Plumier 1980; Denis et al. 1970) and recent single crystal measurements have documented two different anomalies in heat capacity that correlate with magnetic phase transformations (Tsurkan et al. 2003). The complex behavior of thiospinel magnetism results from ferrimagnetic ordering of the Cr and Fe sublattices (Bertinshaw et al. 2014).

Joegoldsteinite is the first known natural occurrence of $MnCr_2S_4$. It is present as two 13-15-µm-size subhedral inclusions in the Social Circle IVA iron meteorite. The meteorite itself was found as a single ~100-kg mass in Georgia, USA in 1926 during ploughing (Buchwald 1975).

The IVA irons constitute the third largest "magmatic" iron-meteorite group; each magmatic group is modeled as having formed by fractional crystallization in the metallic core of a differentiated asteroid (e.g., Scott et al. 1996). IVA iron meteorites are fine octahedrites showing Widmanstätten patterns (Buchwald 1975). The bulk Ni concentrations range from ~60 to ~120 mg/g. Studies of the metallographic cooling rates in IVA iron meteorites have been controversial for several decades (e.g., Willis and Wasson, 1978a,b; Moren and

Goldstein, 1978). Relative to other magmatic irons, the IVA group has large depletions in S, Ga and Ge (Wasson and Richardson 2001). The Ir-rich IVA samples are characterized by lower bulk Ir/Au ratios than comparable members of other iron-meteorite groups (Wasson and Richardson 2001).

The Mn-Cr thiospinel, joegoldsteinite, was approved as a new mineral by the International Mineralogical Association (IMA) in August 2015. It was named in honor of Joseph (Joe) I. Goldstein (1939-2015), Distinguished Professor emeritus of mechanical and industrial engineering and former dean of the College of Engineering at the University of Massachusetts, Amherst. Before arriving at Amherst, Goldstein was the T. L. Diamond Distinguished Professor of Metallurgy and R. D. Stout Professor of Materials Science and Engineering at Lehigh University; he served as vice president for graduate studies and research and as director of Lehigh's Electron Optical Laboratory. Goldstein was well known for his fundamental contributions to research on iron meteorites, metallographic cooling rates, Fe-Ni phase equilibria, electron microscopy and microanalysis.

SAMPLES AND ANALYTICAL METHODS

A polished thick section of Social Circle (TK 724) was made from a 2×3×5-mm-size aliquot from the UCLA meteorite collection. It was examined in reflected light with an Olympus BX60 petrographic microscope and by backscattered-electron (BSE) imaging using a VEGA Tescan SEM at UCLA and a Zeiss 1550VP field-emission SEM at Caltech. Phases were analyzed by energy-dispersive X-ray spectroscopy (EDX) with the SEM and by a JEOL 8200 electron microprobe (EPMA) (WDS mode, 15 kV, 15 nA, focused beam mode using ZAF corrections) at UCLA. The chemical composition is shown in Table 1. A synthesized $FeCr_2S_4$ single crystal, grown by a chemical transport reaction method similar to that of Tsurkan et al. (2001), was used as a standard for S, Cr and Fe measurements.

Single-crystal electron backscatter diffraction (EBSD) analyses at a sub-micrometer scale using methods described in Ma and Rossman (2008, 2009) were performed using an HKL EBSD system on the Zeiss 1550VP SEM at Caltech, operated at 20 kV and 6 nA in focused-beam mode with a 70º tilted stage and in a variable-pressure mode (20 Pa). The EBSD system was calibrated using a single-crystal silicon standard. The structure was determined and cell constants were obtained by matching the experimental EBSD patterns with structures of synthetic $MnCr_2S_4$ and daubréelite.

RESULTS

Petrography and mineral chemistry

Joegoldsteinite occurs as two subhedral inclusions, 13 μm and 15 μm in diameter, in Social Circle thick section TK 724 (Fig. 1). Physical properties were not measured because of the small grain size; however, they are likely to be close to those of daubréelite. Optical properties of joegoldsteinite were assessed in reflected light and compared to daubréelite grains that are adjacent to metallic Fe-Ni in the Aliskerovo and NWA 4704 IIIE iron meteorites (e.g., Breen et al. 2015). Both minerals have similar reflectivity and color. More accurate comparisons could be made if a single section were available that contained grains of both phases. Electron microprobe data indicate that the empirical formula (based on 7 atoms) is $(Mn_{0.82}Fe_{0.23})Cr_{1.99}S_{3.95}$; the general formula is $(Mn,Fe)Cr_2S_4$ and the end-member formula is $MnCr_2S_4$. The calculated density, based on the empirical formula, is 3.71 g cm^{-3}.

Joegoldsteinite is a thiospinel, the Mn analogue of daubréelite ($Fe^{2+}Cr_2S_4$), and a new member of the linnaeite group. In joegoldsteinite and daubréelite, Fe and Mn probably have a +2 valence and occupy the tetrahedral (A) sites. Chromium may have a +3 valence and occupy the octahedral (B) sites. Because joegoldsteinite is Fd3m spinel type, we do not think there is S-S bonding in the structure (a requirement if Cr were +2; McCoy et al., 2014). It thus seems likely that Cr in both daubréelite and joegoldsteinite is located in the octahedral site; Cr3+ should thus be thermodynamically stable at a sufficiently high sulfur fugacity. It seems reasonable that enstatite chondrites could contain both Cr3+ and Cr2+ in different minerals. (Along with nearly pure forsterite and enstatite, some E3 chondrites contain oxidized mafic silicates, i.e., moderately ferroan olivine (Fa11) and low-Ca pyroxene (Fs18) grains; Weisberg and Kimura 2012).

Some tiny grains of joegoldsteinite associated with troilite (FeS) and niningerite ((Mg,Fe)S) were also observed in the Indarch EH4 enstatite chondrite (Fig. 2), but the grains are too small for accurate quantitative analysis by EPMA.

Crystal structure

The EBSD patterns match the cubic space group *Fd3m* spinel-type structure (a = 10.11, V = 1033.4 Å^3, Z = 8) and give a best fit using the $MnCr_2S_4$ structure from Raccah et al. (1966) (Fig. 3), with a mean angular deviation of 0.40º to 0.45º. The cell parameters are taken from data for the matching phase in Raccah et al. (1966). X-ray powder diffraction data (Table 2, in Å for Cu$K\alpha_1$, Bragg-Brentano geometry) were calculated from the cell parameters of Raccah et al. (1966) with the empirical formula, using Powder Cell version 2.4.

DISCUSSION

Other Mn- and Cr-bearing phases in irons and reduced meteorites

The only known phases with detectable Mn in IVA irons besides joegoldsteinite are daubréelite (~0.2-0.8 wt.% Mn) in Maria da Fé (this study) and orthopyroxene (~0.5-0.6 wt.% MnO) and clinopyroxene (~0.5 wt.% MnO) in Steinbach and São João Nepomuceno (Scott et al. 1996).

Social Circle contains a few Cr-rich phases in addition to joegoldsteinite; these include daubréelite ($FeCr_2S_4$), chromite ($FeCr_2O_4$), and possibly, brezinaite (Cr_3S_4) (Buchwald 1975). Additional Cr-rich phases reported in magmatic iron meteorites (but not in the IVA group) include carlsbergite (CrN) in several IIIAB samples and kosmochlor ($NaCrSi_2O_6$) in a few IIA samples (Buchwald 1975).

Enstatite chondrites, such as EH4 Indarch (in which small grains of joegoldsteinite were found), formed under low f_{O2} conditions. These rocks contain mafic silicates (predominantly enstatite, with minor forsterite in unequilibrated samples) with very low FeO, Si-bearing metallic Fe-Ni, and sulfide phases containing cations (e.g., Na, Mg, K, Ca, Ti, Cr, Mn, Fe) that partition mainly into silicates and oxides in more-oxidized assemblages (e.g., Keil 1968; Rubin and Keil 1983; Wasson et al. 1994). For example, sulfide in ordinary chondrites (OC), meteorites that are much more oxidized than enstatite chondrites, is Mn free (e.g., Williams et al. 1985; Rubin et al. 2002); Mn in OC occurs principally in olivine, low-Ca pyroxene, Ca-pyroxene and chondrule mesostasis (e.g., Brearley and Jones 1998).

Additional Mn-bearing sulfides in enstatite chondrites (and related impact-melt rocks and impact-melt breccias) include daubréelite (with 0.7-4.0 wt.% Mn), troilite (FeS: 0.02-0.39 wt.% Mn), oldhamite (CaS: 0.18-1.3 wt.% Mn), niningerite ((Mg,Fe)S: 6.1-12.9 wt.% Mn), keilite ((Fe,Mg)S: 3.4-23.7 wt.% Mn), rudashevskyite ((Fe,Zn)S: 1.6-3.6 wt.% Mn), buseckite ((Fe,Zn,Mn)S, ~10 wt.% Mn), browneite (MnS, ~62 wt.% Mn) and pentlandite ($(Fe,Ni)_9S_8$: 0.66-1.1 wt.% Mn) (Keil 1968, 2007; Lin et al. 1991; Britvin et al.

2008; Ma et al. 2012a,b).

Shock effects

The presence of Neumann lines in Social Circle kamacite indicates that the sample was shocked to at least 10 kb after cooling (Buchwald 1975). A later shock event caused widespread heating of the meteorite: (a) kamacite throughout the mass recrystallized, partially obliterating the Neumann lines (and forming "parallel ghost-lines"), (b) taenite and plessite fields partly decomposed and underwent minor spheroidization, and (c) troilite-metal eutectic shock melts formed (Buchwald 1975). It seems plausible that impact melting of the sulfide assemblages increased the Mn concentration in portions of the S-rich melts, facilitating the crystallization of joegoldsteinite.

After the formation of joegoldsteinite in Social Circle, a minor shock event caused shearing in the grain in the top image of Fig. 1. Displacement by ~0.2 μm occurred along a kamacite grain boundary (also probably produced by shearing) running diagonally from SW to NE-ENE.

Implications

It has been shown that $MnCr_2S_4$ can transform from the spinel structure (where two-thirds of the cations are octahedrally coordinated) to the defect NiAs structure (where all cations are octahedrally coordinated) at temperatures of 1000ºC and pressures of 65 kb (6.5 GPa) (Bouchard 1967). The high-pressure structure is reversible (Vaqueiro et al. 2001). Because of this structural reversibility, empirical observations of thiospinel minerals are unlikely to be useful for constraining the formation temperatures and pressures of asteroidal materials. Nevertheless, chalcospinels, thiospinels and selenospinels have been used for geophysical studies because their phase transitions are good analogs for those of oxyspinel compounds. These are known to show phase transitions in high-pressure regimes (e.g., 29 GPa for $FeCr_2O_4$; Shu et al., 2007), while thiospinel transitions occur at lower pressure (e.g., 9 GPa for $FeCr_2S_4$; Amiel et al. 2011) (Manjon et al. 2014; Santamaría-Pérez et al. 2012).

It seems probable that additional occurrences of joegoldsteinite in enstatite chondrites and IVA irons could be identified by making Mn x-ray maps of enstatite chondrite thin sections and running EDS scans of sulfide grains in sections of iron meteorites. In enstatite chondrites, joegoldsteinite is most likely to be found in association with other sulfide phases; in IVA irons, it could be found as isolated crystals as in Social Circle or as parts of polymineralic sulfide assemblages.

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Table 1. Analytical data for type specimen of joegoldsteinite.

Constituent	wt.%	Range	sd
S	44.3	43.6-44.7	0.35
Cr	36.2	35.7-36.5	0.32
Mn	15.8	15.4-16.0	0.21
Fe	4.5	4.2-5.2	0.30
Ni	0.09	0.02-0.13	0.04
Cu	0.08	0.05-0.11	0.02
Co	<0.03	---	---
total	101.0		

288 **Table 2. Calculated X-ray powder diffraction data for joegoldsteinite (I_{rel} > 1).**

h	*k*	*l*	*d* [Å]	I_{rel}
1	1	1	5.837	18
2	2	0	3.574	34
3	1	1	3.048	100
2	2	2	2.919	2
4	0	0	2.528	58
4	2	2	2.064	12
3	3	3	1.946	3
5	1	1	1.946	50
4	4	0	1.787	95
5	3	1	1.709	3
6	2	0	1.599	4
5	3	3	1.542	12
4	4	4	1.459	10
5	5	1	1.416	4
6	4	2	1.351	7
5	5	3	1.316	11
7	3	1	1.316	5

8	**0**	**0**	**1.264**	**13**
7	**3**	**3**	**1.235**	**1**
8	**2**	**2**	**1.191**	**2**
6	**6**	**0**	**1.191**	**2**
7	**5**	**1**	**1.167**	**8**
5	**5**	**5**	**1.167**	**4**
8	**4**	**0**	**1.130**	**12**
9	**1**	**1**	**1.110**	**2**
9	**3**	**1**	**1.060**	**12**
8	**4**	**4**	**1.032**	**27**
7	**7**	**1**	**1.016**	**1**
10	**2**	**0**	**0.991**	**2**
8	**6**	**2**	**0.991**	**2**
9	**5**	**1**	**0.977**	**17**
9	**5**	**3**	**0.943**	**2**
10	**4**	**2**	**0.923**	**1**
11	**1**	**1**	**0.912**	**1**
7	**7**	**5**	**0.912**	**1**
8	**8**	**0**	**0.894**	**12**

11	**3**	**1**	**0.883**	**1**
9	**5**	**5**	**0.883**	**3**
8	**8**	**2**	**0.880**	**1**
8	**6**	**6**	**0.867**	**2**
9	**7**	**3**	**0.858**	**7**
12	**0**	**0**	**0.843**	**2**
8	**8**	**4**	**0.843**	**9**
7	**7**	**7**	**0.834**	**1**
12	**2**	**2**	**0.820**	**1**
10	**6**	**4**	**0.820**	**9**
9	**7**	**5**	**0.812**	**13**
11	**5**	**3**	**0.812**	**3**
12	**4**	**0**	**0.799**	**40**

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FIGURE CAPTIONS

Fig. 1. Backscattered electron (BSE) images showing two joegoldsteinite grains in Social Circle thick section UCLA TK 724.

Fig. 2. BSE image showing the $(Mn,Fe)Cr_2S_4$ phase in Caltech Indarch section ICM3.

Fig. 3. (left) EBSD patterns of the joegoldsteinite crystals in Figure 1, and (right) the patterns indexed with the *Fd3m* $MnCr_2S_4$ structure.

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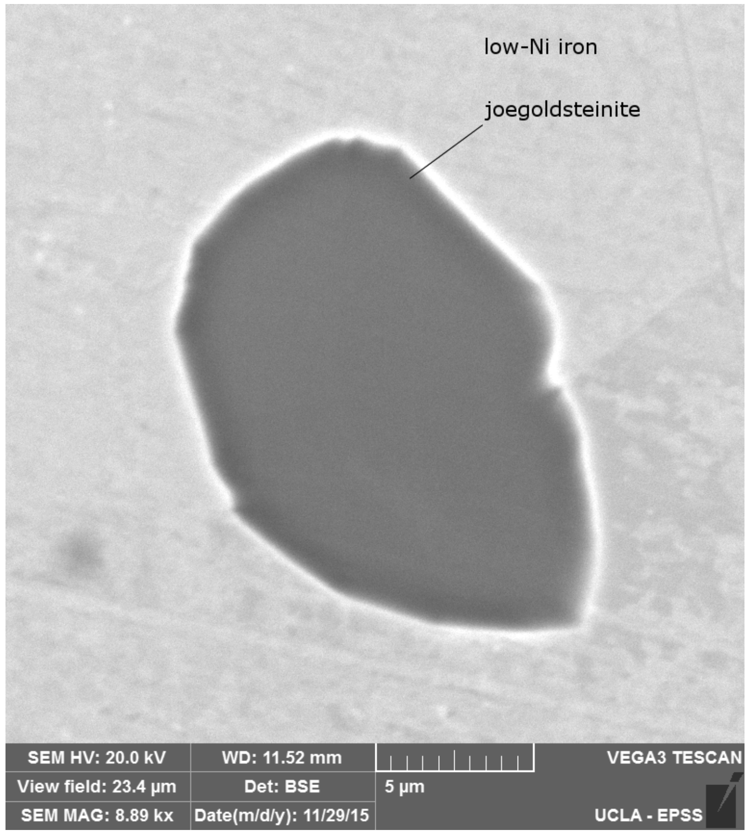

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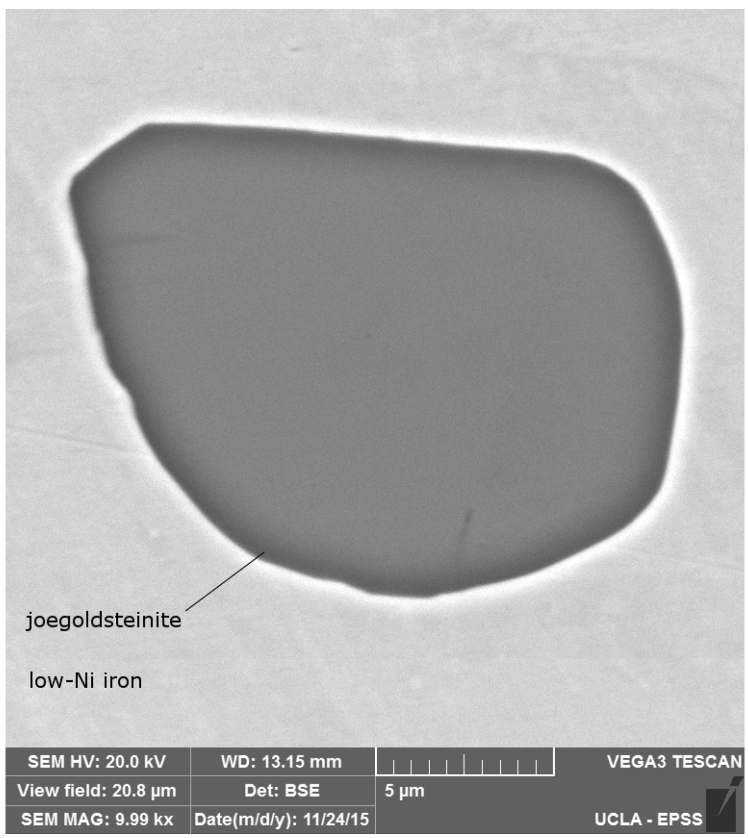

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Fig. 1.

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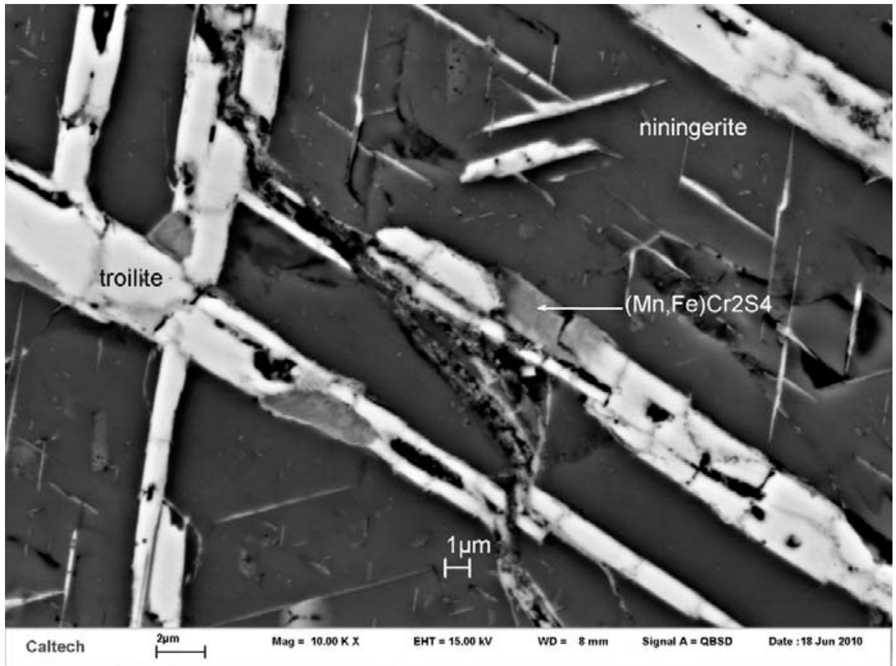
304

305 

306 Fig. 2.

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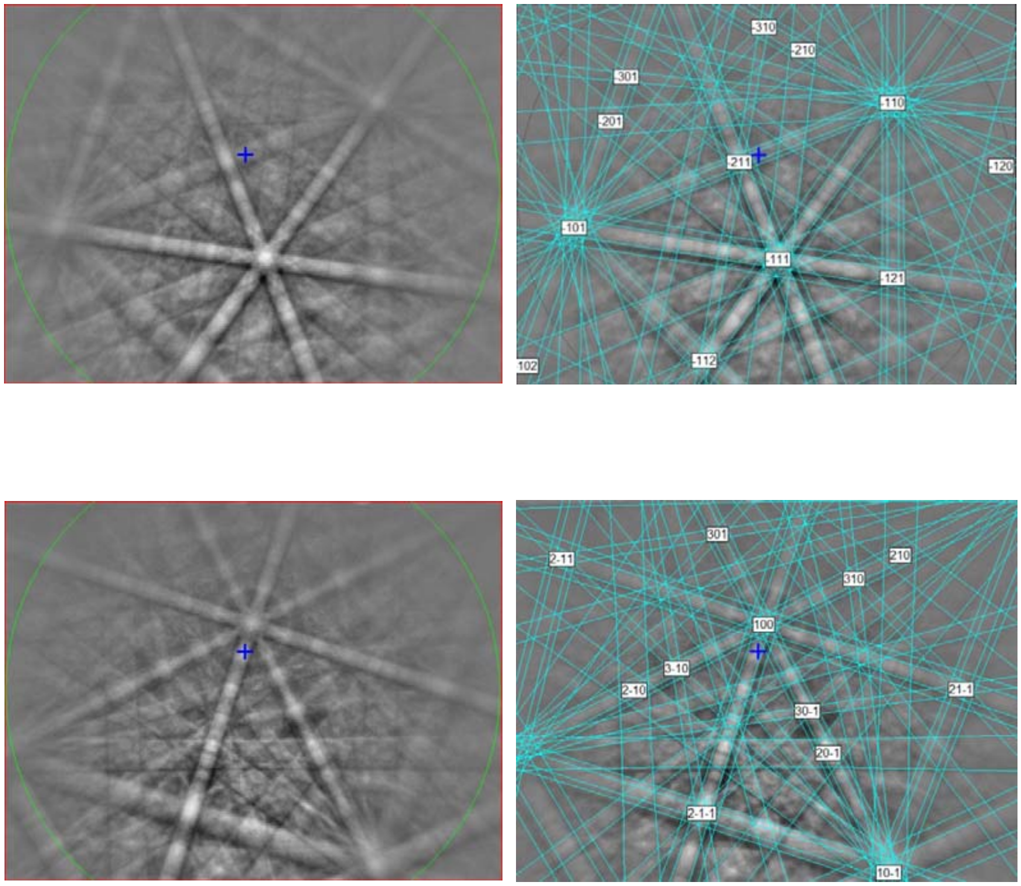

Fig. 3.